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CITRUS CANNERY WASTE, ITS USE AND DISPOSITION

By Harry W. von Loesecke

Bureau of Agricultural and Industrial Chemistry
Agricultural Research Administration
United States Department of Agriculture
Washington 25, D. C.

FOREWORD

This publication is an attempt to bring together in a rather informal manner, published information relative to citrus cannery waste utilization. Treatment of the subject is not intended to be exhaustive.

The use of the waste as a feed has been given considerable space, not only because here, perhaps, is the greatest potential market, but also because published information pertaining to the use of the waste as a feed is widely scattered through the literature.

The historical aspect has in some cases been given in some detail, for it is believed that it may prove to be useful background material for those desiring to make a further evaluation of the problem.

It is hoped that the publication will be helpful to those connected with the citrus industry, and particularly to workers engaged in citrus utilization and waste disposal problems.

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Harry W. von Loesecke
Bureau of Agricultural and Industrial Chemistry
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From 55 to 60% of bulk citrus fruits remain as peel, rag and seeds after processing for juice, concentrates, sections, etc. This waste probably amounts at present to 2.5 million tons annually in the citrus processing regions of the United States. Besides this solid waste, there are over 4 billion gallons of liquid effluents varying in B. O. D. from about 100 to 65,000 p. p. m. These liquid effluents consist of wash water from processing plants, cooling water, floor drippings, exhaust box overflow, press juice, etc. As processing increases, these wastes will increase. Means must be found to either utilize them for the profit of the farmer and processor, or to dispose of them so that they will not constitute a public health nuisance. Much has already been done in utilizing this waste, but considerable remains to be accomplished (1)*.

The composition of the solid waste will vary with the variety of citrus. The composition of grapefruit waste, as determined by Poore (2), is given in Table 1.

TABLE 1.--Analyses of grapefruit cannery waste

	Florida		California
	Peel	Rag	Peel and rag combined
	Percent	*Percent*	*Percent*
Total solids	16.71	15.60	22.02
Ash	0.74	0.75	0.70
Volatile oil	0.43	--	0.56
Acid, as citric	0.74	0.63	0.43
Crude fiber	1.71	1.44	2.00
Crude protein (Nx6.25)	1.13	1.06	1.63
Crude fat (ether extract)	0.28	0.16	0.23
Total sugar (as invert)	6.35	6.30	8.68
Pentosans	0.83	0.44	1.31
Pectin (Calcium pectate)	3.10	3.56	3.93
Naringin	0.40	0.10	0.63

DRYING OF WASTE

In the 1920's and early 1930's of the citrus canning industry in Florida and Texas there was not a sufficient quantity of waste to cause concern. The material was returned to the grove in some instances to be used as a fertilizer, and disced in, or covered with soil to discourage fly breeding. Waste was generally added at the rate of 30 to 40 tons per acre. Use of chemical fertilizers was omitted or used in smaller quantities for some months following the application of citrus wastes.

Another method of utilizing the waste 20 years ago was to add cyanamide to the ground material. From 200 to 400 pounds of calcium cyanamide was added to each ton of ground waste, mixed thoroughly and allowed to stand until the mass felt dry and crumbly. There was a gradual loss of ammonia from the mixture and no guaranteed analysis could be safely given. Sometimes nitrates, ammonium sulfate and superphosphate were added along with the cyanamide (3, 4).

*Numbers in parentheses refer to bibliography at end of text.

With increased canning activities and mounting volumes of waste, it soon became apparent that other means would have to be sought to utilize the material.

As far back as 1923, a California report stated that the feeding of orange pulp to dairy cattle increased the fat content of the milk (5). There is evidence that a rancher, Frank Pellessier near Whittier, California, was feeding fresh orange waste to his dairy herd in 1922.

The fresh material was difficult to handle, it would not keep and rapidly fermented and soured. Drying of the waste would seem a logical solution, but since the material contains from 80 to 85% water, drying would be expensive unless at least part of the water could be removed by means other than by evaporation. Sun-drying was not entirely successful and could not be carried out in every citrus section because of unfavorable weather conditions. The waste has a slimy consistency and initial attempts at pressing to remove part of the water met with failure.

As early as 1927, the California Fruit Growers' Exchange was drying orange pulp as a livestock feed. The material was said to have been limed prior to drying. Dried grapefruit pulp was available in Florida about 1932, and in 1938 Texas was shipping it as far north as New England.

In 1935 Cole and Hall of the California Fruit Growers' Exchange were granted U.S. Patent No. 1,991,242 for a method of disposal of industrial wastes. Their patent covered, among other things, treatment of citrus waste with a pectic enzyme and the addition of lime under specified conditions. Cole and Hall apparently realized the chemical reason for the slimy feel of the waste and knew how to overcome the condition. According to the treatment in their patent, the waste could be mechanically dewatered.

About 1936, limed citrus waste was being dewatered and dried in Florida chiefly as an experimental venture. In 1935, Neal of the University of Florida filed a patent application for a method of treating fruit wastes. The patent was not, however, granted until nearly 16 years later (6). Soon after 1935 there followed a series of patents with the result that even today the patent situation is not clear and the industry has been plagued with a number of patent litigations.

Among the patents issued (other than Cole and Hall's mentioned above) to cover the drying of citrus waste may be mentioned:

U. S. 1,973,084. Food Product and Process for Making. George Thomas Lewis (to Citrus Pulp Corp., Jacksonville, Fla.) Sept. 11, 1934.
U. S. 2,126,947. Stock Food. Emory Cocke, Aug. 16, 1938.
U. S. 2,215,944. Food Product and Process of Making. Daniel Boscawen Vincent, Sept. 24, 1940; Reissue 22,865, April 8, 1947.
U. S. 2,187,501. Treating Citrus Waste Material. A. W. Lissauer (to Louisville Drying Machinery Co.) Jan. 16, 1940.
U. S. 2,362,014. Citrus Feed. A. W. Lissauer and J. Credo (to Citrus Process, Inc.) Nov. 7, 1944.
U. S. 2,455,782. Making Feed Products from Citrus Waste. John M. Kuder. Dec. 7, 1948.
U. S. 2,471,363. Process of Making Food Products. Daniel Boscawen Vincent, May 24, 1949.
U. S. 2,525,645. Method of Processing Citrus Peel and Citrus Peel Liquor. Everette M. Burdick and James S. Allen (to Texsun Citrus Exchange) Oct. 10, 1950.
U. S. 2,536,240. Citrus Pulp Foodstuff. Daniel B. Vincent (to Dan B. Vincent, Inc.) Jan. 2, 1951.
U. S. 2,548,510. Method for Treatment of Fruit Wastes. W. M. Neal (to State of Florida, State Board of Education of Florida, and State Board of Control) Apr. 10, 1951.

The present method of drying the waste consists essentially of adding lime (0.3 to 0.5%) shredding, aging in bins, pressing out part of the water by continuous presses and then drying in either steam tube or direct-fired dryers. It should be pointed out that there is a great deal of variation in plant design and methods used. Some plants add dry lime, while others use a watery slurry. Some operators add lime at a constant rate while others vary lime addition according to the color changes in the aging pulp-lime mixture following shredding. In some instances the limed pulp is aged in bins or pug mills for 45 to 50 minutes, while in other cases aging is for less than 5 minutes. The pulp may or may not be preheated prior to pressing, and in Texas the pulp is seldom, if ever, pressed. The newer plants in Florida also omit pressing, adding dry pulp to the wet pulp in order to prevent sticking in the dryer. Retention time in the dryers will vary from 10 minutes to as much as two hours or more, depending upon the type used. The pulp from the dryers goes to cyclone separators, then to coolers where it is separated into "citrus pulp," "citrus meal," and "fines." Citrus meal is considered to be of lower quality than pulp and sells for a lower price; fines are usually sold as a fertilizer conditioner, or used as fuel at the plant.

Pulp and meal will vary in composition depending upon the fresh waste, degree of pressing, as well as other factors not as yet fully understood. The dried material should not contain more than 10% moisture, and should be protected from insects and rodents. The pulp is somewhat hygroscopic and therefore should be stored in as dry a place as possible (7).

TABLE 2.--Production of citrus pulp, meal, and molasses*
(Expressed in tons)

Season	Florida		Texas		California-Arizona	
	Pulp and meal	Molasses	Pulp and meal	Molasses	Pulp and meal	Molasses
1938-1939[a]	10,600	0	9,555	0	7,003	0
1939-1940[a]	15,250	0	1,950	0	6,061	0
1940-1941[a]	32,730	0	6,150	0	9,237	0
1941-1942[a]	29,697	2,500	6,900	0	17,941	0
1942-1943[a]	43,376	5,700	9,950	0	14,626	0
1943-1944[a]	67,130	44,168	10,050	0	10,421	0
1944-1945[a]	68,724	19,260	16,898	650	15,000	0
1945-1946	108,470	44,168	25,340	2,021	b/ 30,000	NDA
1946-1947	96,914	55,811	21,194	3,267	NDA	NDA
1947-1948	154,181	65,887	31,760	4,675	b/ 35,000	NDA
1948-1949	134,415	41,012	15,237	2,547	b/ 35,000	NDA
1949-1950	163,278	41,689	8,215	250	b/ 40,000	NDA
1950-1951	187,546	70,357	31,333	2,155	b/ 60,000	NDA
1951-1952	218,065	54,035	0	0	b/ 45,000	NDA

a Office of Supply, Commodity Credit Corp., War Food Administration.
b Estimated
NDA No data available.
*One gallon of citrus molasses will weigh between 11 and 12 pounds.

The manufacturer of citrus pulp guarantees an analysis of:

Not less than 6.0% crude protein.
" " " 3.0% crude fat.
" more " 14 to 15% crude fiber.
" less " 53 to 55% nitrogen-free extract.

CITRUS PULP AS A FEED

The use of the dried pulp as a livestock feed was probably first suggested by McDermott, who in 1916 held a Florida Citrus Exchange fellowship at Mellon Institute in Pittsburgh (8). In 1925-26 Scott (9) of the Florida Agricultural Experiment Station at Gainesville reported his work on feeding cattle with dried grapefruit cannery refuse which had been dried experimentally by S. S. Walker in 1925 in the laboratory of the Florida Citrus Exchange. The results of other investigators indicated the suitability of both dried grapefruit and orange pulps as a feed for dairy cattle (10, 11, 12, 13, 14, 15). It has been reported that dried grapefruit peel contains factors which stimulate milk production in dairy cows (14). From reported data it appears that dried grapefruit pulp may stimulate milk production and make for increased yields of butterfat. On the other hand, data available on orange pulp do not seem to indicate this. Probably this difference, if real, would not be of practical importance because the separation of orange from grapefruit wastes in certain producing areas would not be economically feasible. The possibility of imparting flavors to milk by feeding dried citrus pulp to cows has been investigated (16). Dried lemon, grapefruit, and orange pulp fed in quantities up to 4 pounds per cow 1 to 1.5 hours before milking, at which time any flavor that might pass into the milk would be at a maximum, did not impart an objectionable flavor to the milk. When the quantity was increased to 5 pounds undesirable flavors were detected in the milk.

Considerable experimental work has been carried out on the feeding of the dried waste to beef cattle. In 1935 Meal et al (17) of the Florida Agricultural Experiment Station fed dried grapefruit and orange cannery waste to growing heifers. These products were found to be low in crude protein, fiber and fat, but high in nitrogen-free extract (carbohydrates) which was 88 to 92% digestible. Total digestible nutrients per hundred pounds of dry matter were 82.80 and 80.82 pounds for grapefruit and orange, respectively. These feeds are thus placed in the class of high carbohydrate concentrates.

Jones et al (18) at the Texas Agricultural Experiment Station fed dried citrus pulp (grapefruit) to Hereford steer calves replacing part of the ear corn chop with husk with this material. When 25% of the ear corn chop was replaced with dried citrus pulp there were practically equal gains, but slightly higher finish in the citrus fed group. Replacement of as much as 60% of the ear corn chop husk with citrus pulp produced a feed which was less palatable, and had slightly greater laxative effect, and reduced feed consumption, gains and finish. A mixture of 75 parts ear corn chop with husk and 25 parts dried citrus pulp as the carbohydrate concentrate produced satisfactory results.

There have been reports of difficulty in getting animals to consume the dried citrus pulp (12, 13, 17, 18). However, the animals become quickly accustomed to the tast and will then readily consume the material.

Good results have been obtained in the fattening of range cattle on fresh and dried citrus pulp (19). Steers fed dried citrus pulp gained 2.17 pounds per day and required 466 pounds total digestible nutrients for 100 pounds gain.

TABLE 3.--Coefficient of digestibility, and digestible nutrients of dried citrus pulp (17)

	Dried grapefruit pulp			
	Crude protein	Crude fiber	N-free extract	Crude fat
	Percent	*Percent*	*Percent*	*Percent*
Coefficient of digestibility	24.83	71.52	92.43	79.37
Digestible nutrients	1.23	8.54	64.33	0.84
	Dried orange pulp			
Coefficient of digestibility	36.57	93.1	88.51	6.59
Digestible nutrients	2.14	9.99	57.30	0.05

The animals were sleek and glossy and appeared healthy in every respect. Good gains could be secured on an average daily ration of:

5 to 7 pounds of hay
3 pounds cottonseed meal
8 to 9 pounds dried citrus pulp, or
40 to 50 pounds of fresh grapefruit

Fresh grapefruit was readily eaten by the cattle. Well-grown animals can eat whole grapefruit, but for young animals quartering or slicing the fruit is recommended.

CITRUS PULP AS SWINE AND POULTRY FEED

Fresh citrus waste was fed to hogs in citrus producing regions long before the dried material was available. The virtue of the waste for this purpose was the subject of considerable dispute among feeders. There were numerous verbal reports that grapefruit waste caused a bitter flavor in the meat. So far as known, such reports were not substantiated by adequate evidence.

In 1936 Crown and his associates (20) at the Florida Agricultural Experiment Station fed dried grapefruit pulp to pigs. Working with single pigs they were fed dried pulp at 30, 60 and 85% levels. The results showed that successive increments of the pulp made the rations less desirable. As the dried pulp in the ration increased, net yield of dressed pork decreased. Free-choice feeding of pigs resulted in refusal of dried grapefruit pulp.

Fresh grapefruit cannery waste has also been fed to pigs (21). Levels of waste in the feed higher than 5% of the ration (the other constituents being corn and fish meals) reduced the feed intake and rate of gain, increased the feed required per unit of gain and caused more frequent and serious gastric upsets. Self-fed pigs were subject to less frequent gastric upsets than hand-fed. Apparently, self-fed pigs know how much grapefruit they can tolerate.

Feeding of the dried pulp to poultry brought equally unfavorable results. It was found that chicks fed on the pulp (5 to 20% of the ration) showed a decrease in growth during the first 8 weeks (22). Five percent gave good growth, but still below the control group. Mortality in the flock increased as the percent of dried pulp in the ration was increased. Twenty percent citrus pulp in the ration resulted in 100% flock mortality.

Pullets of 8-20 weeks of age, on the other hand, appeared able to utilize citrus meal, based on the rate of gain compared with the controls. There was no relationship between the percent of citrus meal in the ration and pullet mortality.

Laying birds showed no significant difference in number of eggs produced, mortality, or quality of eggs when fed up to 15% dried citrus pulp in the ration.

ENSILAGE OF CITRUS PULP FOR FEED

Bondi (23) constructed cement silos 8 ft. in diameter and 9-1/2 ft. high of which 1-1/2 ft. were under ground. The citrus waste, sliced into 2 in. slices was packed tightly in the silos. The packed material was covered with tar paper, overlaid with straw and about 2 ft. of earth. An anaerobic fermentation takes place during which no butyric acid is formed. The ensiled material contained about 85% water and around 10% free lactic acid (based on the dry matter).

Trench silos were used in other experiments and in some cases Natal grass or sugar cane was mixed with the fresh citrus waste (24). There was some nutrient loss in ensiling, highest losses being in the nitrogen-free extract. Additions of hay or sugar cane increased the efficiency of preserving the nutrients and improved the quality of the silage.

Palatability of citrus pulp silages depended upon previous feeds to which the cattle were accustomed. Generally, citrus silage was quite palatable. The mixtures of citrus with grass hay and cut sugar cane were eaten readily. Plain citrus silage was least palatable of those fed. A feed flavor was transferred through dairy cows into milk when citrus press cake silage was fed 2 hours prior to milking time. Such flavors can be eliminated by proper regulations of feeding time.

CITRUS MOLASSES

As mentioned under the drying of citrus cannery waste, the fresh material is generally pressed after liming and before drying. If not pressed, the limed material is allowed to drain for a short period. In either case, a liquid is obtained containing about 10% solids including approximately 6% sugars. This liquid is unstable, will ferment and create offensive odors. For each ton of dry citrus pulp produced, from 800 to 1,400 gallons of press water will be obtained.

In the early days of drying the pulp, this liquid was disposed of by ponding (this practice still prevails when the citrus molasses market is unfavorable), pumping into deep wells, or returning to groves. None of these methods proved satisfactory. At the present time, most of the material is screened and concentrated in multiple-effect evaporators. The liquid may be flash heated to about 220 deg. F. before concentrating to reduce scaling in the evaporators, particularly in the first effect (25). Clarification of the press liquor prior to concentration gives a better appearing, higher sugar, lower ash, and more stable molasses than that obtained from unclarified press liquor ~~(25)~~. 26

The first citrus molasses plant in the United States was put in operation at Lake Alfred, Florida, in 1941; production was started in Texas in 1945, and at probably the same time in California. Production figures will be found in Table 2.

In Texas, the method of concentration is somewhat different from that in Florida and California. In Texas, the screened liquor is first evaporated in submerged Ozark burner units using natural gas, the first unit concentrating the liquor to 15 deg. Brix, and the second to 22 deg. Brix. The first unit precipitates chiefly calcium citrate and calcium pectinates; the second unit precipitates excess lime as calcium carbonate. The operation of these burners eliminates severe scaling effects in the multiple-effect evaporators (27, 28).

Other than in Florida, citrus molasses is not standardized and lack of wide-spread standards may be the chief deterrant of its use by mixed feed manufacturers (Table 4). Minimum Florida State standards are: it must contain 45% total sugars, as invert, and test not less than 35.5 deg.

Brix by double dilution (29). The product is a thick, dark-brown to almost black liquid with an extremely bitter taste. It is claimed (30) that this bitter taste can be eliminated by treating with water extracted Spanish moss (Tillandsia usneoides) and then with isopropyl alcohol. It is classed as a carbohydrate concentrate.

TABLE 4.--Typical analysis of Florida citrus molasses (29)

Brix deg	72.0	Potassium (K) %	1.1
Nitrogen-free extract	62.0	Calcium (Ca) %	0.8
Total sugars %	45.0	Sodium (Na) %	0.3
Moisture %	29.0	Magnesium (Mg) %	0.1
Reducing sugars %	23.5	Iron (Fe) %	0.08
Sucrose %	20.5	Chlorine (Cl) %	0.07
pH	5.0	Phosphorus (P) %	0.07
Carbonate ash %	4.7	Silica (SiO_2) %	0.01
Acid, as anhyd. citric %	4.5	Manganese (Mn) %	0.008
Nitrogen % x 6.25	4.1	Copper (Cu) %	0.003
Glucoside %	3.0	Niacin (ppm)	35
Pentosans %	1.6	Riboflavin (ppm)	11
Pectin %	1.0	Pantothenic acid (ppm)	10
Fat %	0.2	Inositol	...
Volatile acids %	0.04		
Fiber %	0.00	Viscosity 25 deg. C. centipoises	2000

Referring to Table 4, calculations will reveal that about 10% is unaccounted for. At the present time there are no published data to show of what this unaccounted-for material consists.

Certain changes take place during storage of citrus molasses. Thus, there is a small loss in total sugars; pH decreases, this decrease being greatest in molasses with a pH of 4.5 or below; invert sugar increases; there is an increase in viscosity and precipitation of naringin (31). There may also be spontaneous foaming, the cause of which remains obscure.

CITRUS MOLASSES AS A FEED:

Digestion trials have not been carried out on the molasses (32). The assumption has been made that since citrus molasses resembles beet and cane molasses, the digestive coefficients would be about the same. Applying these figures to the analyses of citrus molasses, give estimated values of 1.4% of digestible crude protein and 56.7% of total digestible nutrients, based on 69.9% of dry matter in the molasses (32).

The molasses has been tried with dairy cattle in a number of ways. Since a 9% level of blackstrap molasses is the amount present in many commercially mixed dairy feeds, Becker et al (33), incorporated 5% citrus molasses into home-mixed concentrates. Twenty-five cows were offered 2 pounds each of the molasses-concentrates after consuming their regular afternoon allowance of feed. All of the feed was consumed. On the second day 10 pounds of citrus molasses were incorporated with 90 pounds of the concentrates and offered in the same manner. The feed was completely consumed by the 25 animals.

When straight molasses was offered to 34 Jersey and Guernsey cows, 26 refused it on the first offering, 17 on the second and 8 on the third. Apparently the bitter taste of the product is involved, for the cows were seen to extend their tongues and twist them from side to side. This action has been interpreted as one of objection against the bitter taste. Other observers say that the material does not taste bitter to cattle, but does taste bitter to horses and mules who will not consume citrus molasses.

Whether or not the bitter taste is objectionable to cattle is probably of little practical importance at present. Nearly every pasture in the cattle raising area of Florida contains

feeding troughs supplied automatically with citrus molasses. Cluster of animals about such troughs is a common sight, and they are apparently allowed to consume the molasses ad lib. It is claimed (34) that it is not unusual for livestock to consume 1/2 gal. per day per animal. Steers have been allowed to consume up to 6 pounds of citrus molasses daily per head for a month without ill effect (35). More recent work on fattening beef calves has been published by the Texas Agricultural Experiment Station (36, 37).

In experiments carried out at the Range Cattle Experiment Station of the Florida Agricultural Experiment Station at Ona, it was found that citrus and cane molasses could be used for either maintenance or fattening and that these products were readily eaten (38). Good gains in range cattle were secured on an average daily ration of 4 to 7 pounds of hay or equivalent pasture, 2 to 3 pounds cottonseed meal and 4 to 5 pounds dried citrus pulp with 4 to 5 pounds of either citrus or cane molasses. Cattle could be maintained throughout the winter on pasture when supplemented with one-half pound of cottonseed pellets and from 3 to 5 pounds of molasses.

In regard to the feeding of citrus molasses to dairy cattle, the effect of the material on milk flavor is of importance. Becker et al (33) obtained milk samples from 4 Jerseys after being in dry lot 10 hours without feed. On other days, citrus molasses was added as 10% of the mixed concentrates fed 2 hours before milking time. The average flavor score* of milk obtained when no feed was consumed by cows 10 hours before milking was only 1.2 points higher than when mixed concentrates containing 10% citrus molasses were fed two hours before milking. The investigators attribute part of this difference in score to the concentrate mixture itself. Some feed flavors were noted, but they were not considered intense or objectionable. It is believed that feeding just after milking should eliminate any feed flavors.

Citrus molasses has been ensiled with Napier grass and with pigeon peas in Florida (33). From 2 to 4% molasses was added to the Napier grass, and 4% molasses with the peas. The palatability of these feeds was observed with 9 Jersey heifers. They showed a decided preference for the molasses-Napier silage.

Recently, experiments have been carried out on feeding hogs with citrus molasses (39, 40). When the molasses was mixed in swine rations it took the pigs from 3 to 7 days to get used to the taste of the molasses in the feed mixture, but after that time they readily ate the ration. Little success met the attempt in getting pigs out on pasture to eat citrus molasses placed alone in an open trough. The hogs would eat the molasses if mixed with ground corn or peanut meal; or if mixed with 50% cane molasses. Since the molasses is not a complete feed in itself, it is recommended that protein supplement and minerals be given. It is also desirable that pigs fed molasses be on lush, green pasture, if possible. It has been estimated (39) that citrus molasses will have a probable range of being worth somewhere between 70 to 90% the feeding value of corn.

Some producers of pulp and molasses add part of the molasses back to the pulp. This may be done before or after drying the pulp and the amount of molasses added will vary from 10 to 35%. In some instances the dried pulp with added molasses is pelleted. Some producers sell the molasses added product as such, while others make no declaration. Very few, if any, declare molasses addition on the tag attached to the bag of feed. The addition of molasses will, of course, alter the composition of the finished feed, particularly an increase in the nitrogen-free extract.

Florida citrus molasses producers are now adding urea to the product, in some cases, to increase the nitrogen content and enhance its feed value. There have been reports from Texas that the pulp is being ammoniated with fertilizer grade anhydrous ammonia (41). It is claimed that ammoniation increases the nitrogen content by 5 to 6%, equivalent to a crude protein content of about 40%. Ammoniation of agricultural materials is the subject of at least one patent (42).

*Scored according to American Dairy Science Assn. score card which allows 45 points for flavor.

OTHER USES FOR PRESS LIQUOR AND CITRUS MOLASSES

Since the press liquor and molasses contain sugars, most of which are fermentable, these could, theoretically, serve as the raw material for a number of products, such as citric acid, alcohol, yeast, lactic acid, glycerin, vitamin B_{12}, butyl alcohol, acetone to mention a few. Only three of these have been given serious consideration. A recent patent (43) describes techniques for obtaining yeast, alcohol, citric acid and a dry residue from the press liquors.

FEED YEAST:

In 1942 Nolte and co-workers (44) showed that feed yeast and alcohol could be prepared from the press liquor. Using a pure culture of Torula utilis, yields ranging from 37 to 48% of dry yeast (based on the total sugar content of the liquor) were obtained. These workers utilized batches of 2 to 9 gallons of the liquor.

Later Veldhuis and Gordon (45) produced feed yeast on a pilot plant scale from the liquor. Using continuous propagation with a maximum feed rate of 185 gallons per hour, they obtained yields of from 33 to 60% based on the sugars present.

At the present time (1953), there is no commercial production of feed yeast from press liquor. Such production is a matter of economics which has been the subject of considerable dispute. Primary grown yeast on citrus molasses would have to compete with brewers' dry yeast (of which there is an annual production of some 25 million pounds), which can be produced cheaper, because it is a byproduct from beer making; yeast from sulfite waste liquor, vitamin B_{12}, oilseed meals, and distillers' solubles. The market for feed yeast at the present time is limited and highly competitive (46).

Alcohol production from the press liquor and molasses is carried out in Florida at Lake Alfred in a plant having a capacity of about 10,000 gallons per day. The plant appears to operate spasmodically, according to the alcohol market. There is also some alcohol production in California.

Lactic acid is produced commercially by the fermentation of a sugar solution, usually molasses, skim milk, buttermilk and whey by inoculating these media with a suitable strain of a lactic acid organism. Annual production of the acid in the United States amounts to about 8.0 million pounds. Probably industry could absorb a great deal more than this, if the price could be reduced. Its chief use is in foods, it also finds use in the leather industry and in the plastics field, especially in making cast plastics.

Lactic acid has been prepared on a semi-plant scale in Florida. Citrus molasses (as well as the press liquor) was used as the raw material, and the acid obtained through an ester of the acid (47, 48, 49). The Florida plant is no longer in operation.

Naringin is the bitter glycoside present in grapefruit. The name is derived from the sanskrit "narengi" meaning "orange," and Hoffman applied the name to the glycoside he and DeVry investigated.

Naringin can be prepared from the press liquor (provided it has been obtained chiefly from grapefruit) by permitting it to stand in a cool place. It can more conveniently be prepared by leaching of ground grapefruit peel.

Upon hydrolysis with dilute acid, naringenin, glucose and rhamnose are obtained (50). Yield of rhamnose amounts to about 20% of the naringin, or 62% of the theoretical. The market for rhamnose is limited to chiefly bacteriological work.

When naringenin is hydrolyzed with concentrated potassium hydroxide, p-coumaric acid and phloroglucinol are formed. The market for both of these products is limited; they find use chiefly as analytical reagents and in organic synthesis.

Naringin, itself, has a limited market. It has been used in the preparation of grapefruit beverages to impart a bitter taste, and in preparing certain marmalades of the bitter type. It has recently been reported (51) that 6,000 pounds annually of naringin is being produced in Arizona and exported for beverage purposes.

Limonene (stripper oil). In flash heating the press liquor prior to concentration for molasses production, d-limonene is recovered. The material has essentially the following properties (29).

Sp. Gr. at 25 deg. C.	0.8398 - 0.8443
Optical rotation $(\alpha)^{D}$ 25 deg. C.	+ 95.55 deg. - + 98.90 deg.
Refractive Index n_D 25 deg. C.	1.4713 - 1.4721
Ester content	0.07 - 2.46%
Aldehyde (as decyl aldehyde)	0.47 - 1.50%
Residue on evaporation	0.03 - 0.79%

It is colorless liquid, with a characteristic odor suggestive of lemons, and when applied to the skin has a burning sensation. The product is not pure d-limonene, since the pure compound has an optical rotation of around +124 deg. Limonene belongs to an interesting class of compounds called terpenes, and there are three limonenes known: d-limonene, 1-limonene and dl-limonene (dipentene) which is optically inactive. The reader desiring further information should consult: "The Terpenes," Vol. 1 (1947), by J. L. Simonsen, published by Cambridge at the University Press, Cambridge, England.

At the present time use of d-limonene (stripper oil) from citrus is limited. Some of the Florida production is sold to plastic manufacturers, and some is also said to be used for the preparation of isoprene. A patent (52) makes use of d-limonene in the preparation of penetrating oils. The oil may have possibilities in the preparation of fine organic chemicals (53), but would have to compete with cheaper dipentene from pine wood waste.

"Vitamin P." This flavone, originally named by Szent-Györgi, has lost favor as a vitamin, but reports continue to suggest that it may have some important pharmacologic properties. There is limited production of so-called vitamin P in Florida, citrus molasses serving as the raw material. The crude material is dark brown in color with a sweet odor and bitter taste.

OTHER PRODUCTS FROM THE PEEL

Peel oil is produced in practically every citrus growing country of the world. Methods of production will generally vary with the region.

In the United States, California produces lemon, orange and grapefruit oils; Texas, grapefruit oil; and Florida, orange, grapefruit and small amounts of tangerine and lime oils.

Yields of domestic cold pressed oil will vary with method of extraction, season, region, variety as well as other factors. Yields are approximately as follows, per ton of peel:

orange	1.5 to 9.5 pounds
grapefruit	1 to 2 pounds
lemon	10 to 12 pounds
lime	0.1 to 0.3 pounds
tangerine	2 to 4 pounds

Physical constants of domestic citrus oils are given in Tables 5 and 6. For a comprehensive study of commercial production methods and properties of citrus oils, the reader should consult the following:

The Essential Oils, Vol. 3. Ernest Guenther.
D. Van Nostrand, Inc., New York, N. Y. (1949).
Florida Citrus Oils. J. W. Kesterson and O. R. McDuff.
Florida Agr. Exp. Sta. Bul. 452 (1948).
Oxidative Stability of Orange Oil as Related to Method of Extraction. J. W. Kesterson and R. Hendrickson.
Am. Perf., 57, 441-444 (1951).
Analyses and Composition of California Lemon and Orange Oils.
H. D. Poore. U. S. Dept. Agr. Tech. Bul. 241 (1932).
Deterioration of Orange Oil. H. Flores and R. E. Morse.
Food Technol., 6, 6 (1952).

Besides cold pressed oils, distilled oils are prepared by steam distilling the pomace left from the cold pressed oil. Distilled lemon oil is made in California also by steaming lemon juice prior to citric acid recovery. Distilled oils differ in composition from cold pressed, and they also sell for a lower price. Terpeneless and sesquiterpeneless oils (sometimes called "concentrated oils") are those from which the terpenes and sesquiterpenes have been removed, respectively. These two components are prone to oxidation and other deteriorative changes, and their removal should make such oils more stable. These types of oil are not generally made by original producers of cold pressed oil because removal of terpenes requires close technical control and more expensive equipment than for the recovery of cold pressed oils. Terpeneless oils are therefore produced by the larger oil supply houses. For information relative to methods of preparing terpeneless oils, the reader should consult Guenther's "The Essential Oils," Vol. 1, D. Van Nostrand, Inc., New York, N. Y. (1949).

The market for citrus oils is competitive and limited, and prices fluctuate widely. The oils are used primarily for flavoring baked goods, confectionery and beverages, and to a lesser extent in the preparation of certain perfumes.

Pectin. Citrus pectin is prepared from the peel (orange, lemon, grapefruit), production at present (1953) being limited to California. A few years ago there was production in Texas, and during World War II, in Florida.

From 4 to 5 million pounds of pectin are produced annually in California, about 100,000 tons of fresh peel being used for this purpose. Pectin is extracted from the peel by leaching with a dilute acid solution, the pectin being recovered from the dilute acid solution either by precipitation with alcohol (usually isopropyl) or as an aluminum complex with aluminum chloride and sodium carbonate, or aluminum chloride and ammonium hydroxide. The complex is further treated to recover the pectin. The process of recovering citrus pectin is covered by several patents, some of which have expired.

Pectin is sold on the basis of grade, such as, for example, 150 grade. This means that one pound of such a pectin will set to a gel 150 pounds of sugar under proper acid conditions. Pectin is used chiefly in the preparation of jams and jellies. Present production probably takes care of consumer demands, and research is being conducted toward new uses and lower production costs.

Ordinary pectin used in the manufacture of jams and jellies requires the use of approximately 65% sugar and small amounts of acid to form a stable gel. By changing the structure of the pectin molecule, pectins may be obtained which require only calcium, for instance, to form stable gels. Such pectins are called "low methoxyl pectins" in comparison to ordinary pectins which have a higher methoxy content in the molecule.

TABLE 5.--Approximate physical constants of domestic cold pressed citrus oils
(Data from various sources)

Source	SP. GR. at 25° C.	Refractive index $N_D 20°C.$	Optical rotation (α) D 25°C.	10% Distillate Refractive index $N_D 20°C.$	10% Distillate Optical rotation (α) D 25°C.	Residue on evaporation %
California	0.843-0.846	1.4731-1.4742	+98.33°-+94.0°			3.5-5.1
California (Valencia)	0.8440	1.4735	+97.78°	1.4723	+99.21°	3.6
California (Navel)	0.8445	1.4738	+96.93°	1.4724	+98.71°	4.53
Florida orange	0.8416-0.8458	1.4718-1.4734	+95.16°-+97.76°	1.4703-1.4715	+96.81°-98.70°	1.07-4.93
Florida grapefruit	0.8508-0.8532	1.4746-1.4761	+91.19°-+92.96°	1.4698-1.4712	+95.81°-98.14°	6.02-8.02
Florida tangerine	0.8456	1.4734	+91.18°	1.4711	+92.68°	4.53
Florida lime	d 0.881	1.4849	d +40.55°	--	--	13.74
California lemon	0.8475-0.8525	1.4738-1.4749	+52.71-+70.18	1.4726-1.4737	+46.47°-+65.74°	2.01-4.52
Texas grapefruit (Marsh seedless)	d 0.856	1.4769	d +96.45	--	--	--
Texas orange (Valencia)	d 0.846	1.4742	d +97.10	--	--	--

d 20°C.

TABLE 6.--Approximate physical characteristics of domestic steam distilled citrus oils (29).

Source	SP. GR. 20° C.	Refractive index $N_D 20°C.$	Optical rotation (α) D20°C.	Aldehydes %	Esters %	Residue on evaporation %
California orange	0.840 0.842*	1.4717-1.4730	+99.1°*	--	--	0.4-1.0
Florida Grapefruit**	0.8415-0.8439	1.4714-1.4746	+91.50°-+96.50°	2.30-4.06	0.08-2.52	0.19-3.66
Florida tangerine	0.8407	1.4720	+93.67°	1.24	0.25	0.20
Florida lime (Persian)	0.8556-0.8579	1.4743-1.4751	+46.84°-+50.52°	1.61-2.71	2.41-3.49	0.18-1.23

*At 25°C.
**Vacuum steam-distilled.

Use of low methoxyl pectins results in the saving of sugar, and they can be used for coating food materials for protective purposes; for preparing certain types of pudding powders and other specialized uses. The following publications will be found useful to those desiring further information regarding low-methoxyl pectins:

Gelation Characteristics of Acid-Precipitated Pectinates. Harry S. Owens, Roland M. McCready and W. Dayton Maclay. Food Tech., 3, 77 (1949)

Effect of Methyl Ester Content of Pectinates Upon Gel Characteristics at Different Concentrations of Sugar. George L. Baker and Marvin Goodwin. Univ. Del. Agr. Exp. Sta. Bul. 246 (1944)

Demethylation of Pectin and Its Effect on Jellying Properties. G. L. Baker and M. W. Goodwin. Univ. Del. Agr. Exp. Sta. Bul. 234 (1941)

Low Sugar Jellying Pectinates. C. H. Hills, J. W. White and G. L. Baker. Proc. Inst. Food Tech., 1942, 47

Alkali-hydrolyzed Pectins are Potential Industrial Products. R. M. McCready, H. W. Owens and W. D. Maclay. Food Ind., 16, 794 (1944)

Effect of Methoxyl Content of Pectin on the Properties of High-Solid Gels. H. W. Owens and W. D. Maclay, J. Colloid Sci., 1, 313 (1946)

Pectinate Films. T. H. Schultz, H. S. Owens and W. D. Maclay. Ibid., 3, 53 (1948)

Use of Polymetaphosphates and Polyphosphates in the Extraction of Pectin and Pectinic Acids from Citrus Peel. R. M. McCready, A. D. Shepard and W. D. Maclay. Fruit Prod. J., 27 (2) 36 (1947).

The Pectic Substances. Z. I. Kertesz. Interscience Publishers, Inc., New York, N. Y. 1951

Low methoxyl pectins are in commercial production, but they cost somewhat more than ordinary pectin. However, they have a large potential use in producing low-sugar gels, reducing "run off" in certain frozen fruits, coating candied fruits and chocolate bars, and for many other uses.

Crude Pectin was a World War II venture when the demand for pectin in the United States and Allied Nations exceeded the expanded capacity of the pectin industry. There was also limited production in Florida during 1948 or 1949, the crude pectin being sold at $1.00 per ton per grade of pectin. The crude pectin generally ran around 30 grade. In reality this is not crude pectin, but merely leached grapefruit peel, but the trade considered it as a crude pectin.

The method for preparing this crude pectin was worked out by Pulley and his co-workers (54). Enzymes in fresh grapefruit peel are first inactivated by boiling the ground peel in water, and then most of the soluble material removed with water. The peel is then washed with cool water, pressed to remove excess of water and then dried to 4 to 8% moisture content.

The jelly maker would extract the pectin from this material by means of hot acid solutions, the acid extract thus obtained being used in the preparation of jams, jellies and marmalades.

Hesperidin is a glycoside found in oranges. It is obtained in California from orange peel by extracting with an alkali and then acidifying the alkaline extract with hydrochloric acid. The precipitated glycoside is purified by recrystallizing from sodium hydroxide and isopropyl alcohol or from pyridine.

Hesperidin is converted to the chalcone by treatment with alkali and methylated to form hesperidin methyl chalcone. This product is completely soluble in water and is stable under ordinary conditions of acidity and temperature. Hesperidin and its chalcone have "vitamin P" activity and thus find use in therapeutics. Hesperidin methyl chalcone is prepared in California, and is an article of commerce. Considerable research has been conducted on the nature of "Vitamin P" but much more needs to be accomplished.

Brined and Candied Peel. Citrus peel (grapefruit, orange, lemon and whole citron) is preserved in brine and subsequently used in the preparation of candies and mincemeat. The brining technique has long been established on the continent, but is a comparatively new industry in the United States.

In the brining of peel, material is selected that is free of scale or melanose. The peel generally consists of "cups" left after reaming to obtain juice. The cleaned cups are packed by nesting into 52 gal. fir, paraffined lined barrels with a 10% salt solution in which they are allowed to cure. Properly cured peel is firm and has a translucent appearance. After curing, the peel is stored and shipped in a 2-15% brine. Sulfur dioxide is also sometimes added.

Candied citrus peel is prepared from either the fresh peel, or from washed brined peel. Candied peel is a specialized industry usually carried on by small concerns and in a great many instances sales are direct to the consumer. Considerable hand labor is involved in the candying process, and the finished product is sold for a high price which some believe not to be in keeping with its epicurean qualities. Several methods are used for candying the peel, but in general may be classified as the "rapid process"

and the "slow process." The latter technique is now used very little because it requires a considerable investment in sugar, treatment vessels, space and labor. Both methods consist of impregnating the peel with sugar sirup until the sugar concentration is high enough to prevent spoiling. Following impregnation the peel is drained and dried. In some instances the material is dyed with certified food colors. The following references will give further details relative to the candying of peel:

Commercial Fruit and Vegetable Products. W. V. Cruess, 3rd Ed. 1948, p. 423-424. McGraw-Hill Book Co., Inc., New York, N. Y.

Observations on Brining and Candying of Citron Peel. W. V. Cruess and D. Glickson. Fruit Products Journ. September 1932.

The Preparation of Candied Peel. J. Elsbury. Food Manuf., 7, 237 (1932).

Outlines of Food Technology. Harry W. von Loesecke, 2nd Ed. 1949, p. 498-500. Reinhold Publishing Corp., New York, N. Y.

Citrus Fruit Products. E. M. Chace, H. W. von Loesecke and J. L. Heid. U. S. Dept. Agr. Circ. 577 (1940).

CITRUS SEED OIL

The seeds of citrus fruits have long been recognized as a potential source of oil, but their commercial utilization in this respect has been governed by the availability of the seeds, as well as the oil market situation, and use of the seed oil plant for some other oil or oils when citrus seeds are not available. Drying and storing the seeds for off-season use has not been considered practical; the oil in stored seeds will increase in free fatty acid content.

Citrus seeds cannot serve as a basis for oil recovery except as they are a byproduct in the processing of juice or other citrus products.

Domestic commercial production of grapefruit seed oil was first attempted in Florida during 1938-39, at which time approximately 90,000 pounds of crude oil were produced and then the industry died. During World War II, interest in seed oil was revived, but production was not great. Around 1946 or 1947 there were two producers of oil in Florida: One at Lakeland and the other at Plymouth. It is estimated that about 700,000 pounds of citrus seed oil were produced during the 1948-49 season, and probably more than this during the 1950-51 season. Seeds are separated and collected at the canneries. Originally, the seeds were allowed to undergo a fermentation to loosen the slimy coating and adhering pulp (55). In present production methods, excess pulp is separated from the seeds by using a paddle-type finisher or similar piece of equipment. The cleaned seeds are dried, tempered and then pressed without decortication, in expeller type presses. Usually, no effort is made to separate the seeds from different varieties of citrus, although chemical and physical properties of the oils will differ (Table 7).

Crude citrus oils are bitter tasting, but can be refined to yield bland, light-colored oils. The bitter taste in grapefruit seed oil may be due to limonin (55). Unless winterized, the oils solidify around 10 to 14 deg. F., an important consideration in shipment.

So far as known, these oils have been used for industrial purposes, chiefly in the leather and textile fields (56, 57). There has been a small use in cooking oils.

The press cake obtained after extraction of the oil contains from 5 to 7% residual oil. Solvent extraction of the seeds would result in greater oil yield, but there are no published data to indicate the feasibility of solvent extraction. An analysis of the press cake is given in Table 8.

TABLE 7.--Chemical and physical characteristics of domestic expelled citrus seed oils

	Grapefruit seed*	Orange seed**	Tangerine seed***
	(Commercially produced in Fla.)	(California Valencia; experimental)	(Florida Fancy; experimental)
Sp. Gr.	0.9197	0.9153	0.9168
Refractive index $N_D 20^{\circ}C$	1.4698	1.4686	1.4698
Acid value	0.95	5.9	4.83
Saponification No	193.0	197.5	193.8
Iodine No	100.9 (Hanus)	101.7 (Wijs)	107.4 (Wijs)
Unsaponifiable matter %	0.48	0.95	0.34
Acetyl value	2.4	--	--
Reichert Meissl value	0.47	--	--
Polenski value	0.20	--	--
Moisture and volatile matter %	0.13	--	--
Meal content %	0.05	--	--

*Nolte, A. J., and von Loesecke, Harry W. Ind. Eng. Chem., 32, 1244 (1940)
**Van Atta, G. R., and Dietrick, W. C. Oil and Soap. 21, 19 (1944)
***Swift, L. J., J. Am. Oil Chemists' Soc., 26,438 (1949)

TABLE 8.--Composition of citrus seed oil meal
(From Plymouth Citrus Growers Assn., Plymouth, Fla.)

	Percent
Moisture	6.3
Crude protein	32.50
Fat	'7.5
Crude fiber	8.20
Ash	7.50
Potash	1.40
Calcium	5.75
Magnesium	0.46
Phosphorus	0.13
Iron	0.031
Copper	0.0007
Zinc	0.0008
Manganese	0.0008
Boron	0.0032

The press cake can be returned to the citrus pulp. Driggers et al (58) have fed the press cake to growing chicks with unfavorable results. These workers believe that the meal contains a toxic principle, probably limonin. The meal can be detoxified by extraction first with ether, then with acetone and finally with alcohol. Commercially, such a procedure would seem expensive, for solvent loss, especially in the case of ether would be rather high. Expeller processed citrus seed meal has been found to be unpalatable for the pig even when fed at 10% levels in the ration; when fed above this level it is harmful (59).

Because of the low production of seed oil, disposal of the press cake is not at present of importance.

DISPOSAL OF CANNERY EFFLUENTS

The volume and composition of effluents from citrus canneries, consisting of floor washings, cooling water, drippings, exhaust box overflow, etc., will vary according to the size of the cannery and kind of citrus pack being made. (Table 9) Volume is about from 50 to 56.5 gallons per case of No. 2's.

TABLE 9.--Analyses of screened Florida cannery effluents (60)

Type of pack	Plant No. 1 sections and juices	Plant No. 2 sections and juices	Plant No. 3 sections and juices	Plant No. 4 sections and juices
Total solids*	1403	4084	672	3550
Organic solids*	1189	3633	551	3246
Inorganic solids*	214	451	121	304
Suspended solids*	61	248	171	287
pH	7.2	6.1	6.5	5.6
5 day B.O.D.*	583	2000	343	1722

*Parts per million.

Proper disposal of these effluents has been given a great deal of thought. It has been shown (60) that trickling filters are satisfactory, but investment costs would run high. At the present time, however, some canning plants in Florida are experimenting with trickling filters. Satisfactory results are reported by inoculating citrus cannery effluents with bakers' yeast and allowing to ferment (61). Such a procedure called for a retention period of 48 hours, a practice which would not be universally feasible because of lack of space. Considerable research is being devoted to anaerobic fermentation of the waste to yield methane (62, 63), and work is being carried on in the Rio Grande Valley of Texas by public health agencies relative to different means of treating citrus cannery effluents (64, 65).

At the present time, there is no entirely satisfactory method of disposing of these effluents, and the problem is one of vital public health importance. Effluents are now disposed of by:

1. Emptying into lakes. This causes pollution and destruction of fish life, especially when the body of water is of insufficient area to dilute the waste.
2. Handling by city sewerage system. This has proven satisfactory where capacity of the system is sufficient to handle the additional load. In some instances there has been damage to pumps and piping, clogging of sand beds and excess foaming in Imhoff or primary settling tanks.
3. Primary settling. Retention time is generally too short to accomplish much of value. Effluent from primary settling tanks may flow over weirs in the attempt to aerate. Where effluents from settling tanks are discharged into bodies of water, there is a build-up of sludge at the outfall and generally considerable gassing.
4. Flooding of waste lands. This method has been satisfactory where sufficient areas are available, there is a low water table, and the areas are far enough from human habitation.
5. Tide-water rivers. This practice is limited to canneries located reasonably near the coast. It has not been entirely satisfactory because of sludge formation at the outfall, and danger of polluting shellfish beds.

Under no circumstances can effluents from canneries or retention tanks be discharged into a stream without permission from the proper Federal, State or local authorities.

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